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Indian Standard SPECIFICATION FOR p-NITROANISOLE

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INDIAN STANDARDS INSTITUTION
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Indian Standard

SPECIFICATION FOR p-NITROANISOLE

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(Continued on page 2)

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IS: 6260 - 1971

(Continued from page 1)

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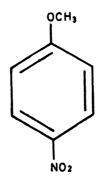
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Indian Standard

SPECIFICATION FOR p-NITROANISOLE

0. FOREWORD

- 0.1 This Indian Standard was adopted by the Indian Standards Institution on 23 August 1971, after the draft finalized by the Dye Intermediates Sectional Committee had been approved by the Chemical Division Council.
- **0.2** p-Nitroanisole ($C_7H_7NO_3$) is used in the manufacture of p-anisidine. It has the following structural formula:



p-Nitroanisole
(Molecular weight=153)

- 0.3 This standard is one of a series of Indian Standards on dye intermediates. A complete list of such standards is given on P 6.
- 0.4 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS: 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

^{*}Rules for rounding off numerical values (revised).

18: 6260 - 1971

1. SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for p-nitroanisole.

2. REQUIREMENTS

- 2.1 Description The material shall be in the form of a light brown or brownish-yellow crystalline fused mass.
- 2.2 The material shall also comply with the requirements given in Table 1.

TABLE 1 REQUIREMENTS FOR p-NITROANISOLE METHOD OF TEST (REF SL CHARACTERISTIC REQUIREMENT No. TO CL No. IN IS: 5299-1969*) (1)(2)(4) (3) i) Crystallization point, °C 51.2 to 52.0 7.3.2 ii) Nitro compound content, 98.5 14 percent by weight (expressed as C₇H₇NO₈, mol wt 153), Min

3. PACKING AND MARKING

- 3.1 Packing Unless otherwise agreed to, the material shall be packed in suitable drums (see IS: 2552-1963*).
- 3.2 Marking The containers shall be securely closed and shall bear legibly and indelibly, the following information:
 - a) Name of the material;
 - b) Name of the manufacturer and his trade-mark, if any;
 - c) Lot or batch number; and
 - d) Tare, net and gross weights.
- 3.2.1 The containers may also be marked with the ISI Certification Mark.

NOTE — The use of the ISI Certification Mark is governed by the provisions of the Indian Standards Institution (Certification Marks) Act, and the Rules and Regulations made thereunder. Presence of this mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard, under a well-defined system of inspection, testing and quality control during production. This system, which is devised and supervised by ISI and operated by the producer, has the further safeguard that the products as actually marketed are continuously checked by ISI for conformity to the standard. Details of conditions, under which a licence for the use of the ISI Certification Mark may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution.

^{*}Methods of sampling and tests for dye intermediates.

^{*}Specification for steel drums (galvanized and ungalvanized).

4. SAMPLING

- 4.1 The material shall be melted and sampled in accordance with the method prescribed in 3 of IS: 5299-1969* using a sample bottle or can as necessary.
- **4.2 Number of Tests** Nitro compound content and crystallization point of each individual sample shall be determined separately.
- 4.3 Criteria for Conformity The lot shall be declared as conforming to the standard if the test results as obtained in 4.2 satisfy the corresponding requirements given in Table 1.

^{*}Methods of sampling and tests for dye intermediates.

INDIAN STANDARDS

ON

Dye Intermediates

| IS: | | | | | | | Rs |
|-------------------|-----------------------------|-------------|-----------------|-------------|-----|-----|--------------|
| 2630-1964 | Nitrobenzene, technical | ••• | ••• | ••• | ••• | ••• | 3.00 |
| 2740-1964 | Sulphanilic acid, technic | al | ••• | ••• | ••• | ••• | 3.00 |
| 2741-1964 | β-naphthol | ••• | ••• | ••• | ••• | ••• | 3.20 |
| 2744-1964 | α-naphthylamine | ••• | ••• | ••• | ••• | | 3.00 |
| 2833-1964 | Aniline, technical | ••• | ••• | ••• | ••• | ••• | 4.50 |
| 3229-1965 | Naphthionic acid (sodiun | asalt) | ••• | ••• | ••• | ••• | 2.50 |
| 3242-1965 | β-oxy naphthoic acid (b | on acid) | ••• | ••• | ••• | ••• | 3.00 |
| 3562-1965 | p-nitrotoluene, technical | ••• | ••• | ••• | ••• | ••• | 4.50 |
| 4265-1967 | 4-4' Diaminostilbene 2-2' | disulphoni | ic ac id | ••• | ••• | ••• | 4.00 |
| 4334-1967 | o-chloroaniline | ••• | ••• | ••• | ••• | ••• | 5 ·00 |
| 4335-1967 | m-chloroaniline | ••• | ••• | ••• | ••• | | 5 ·00 |
| 4336-1967 | p-chloroaniline | ••• | ••• | ••• | •• | ••• | 5 ·50 |
| 4425-1967 | p-Nitrotoluene e-sulphonie | c acid | ••• | ••• | ••• | ••• | 4.00 |
| 4523-1968 | Acetoacetanilide | ••• | ••• | ••• | ••• | ••• | 4.00 |
| 4524-1968 | Acetoacet-o-chloroanilide | ••• | ••• | ••• | ••• | ••• | 4.00 |
| 4525-1968 | p-Aminoacetanilide | ••• | ••• | ••• | ••• | ••• | 4:00 |
| 4526-1968 | 2, 5 Dichloroaniline | ••• | ••• | ••• | ••• | ••• | 5.00 |
| 4527-1968 | 2-Nitro-4-chlorotoluene | ••• | ••• | ••• | ••• | ••• | 5.00 |
| 4528-1968 | 4-4'-Dinitrostilbene-2-2'-c | lisulphonic | cacid (disc | dium salt) | | ••• | 5.00 |
| 5042-1969 | 1-Aminoanthraquinone | ••• | ••• | ••• | ••• | ••• | 4.00 |
| 5043-1969 | 2-Aminoanthraquino ne | ••• | ••• | ••• | ••• | ••• | 4.00 |
| 5044- 1969 | Benzanthrone | ••• | ••• | ••• | ••• | ••• | 5.00 |
| 5045-1969 | Metanilic acid, technical | ••• | ••• | ••• | ••• | ••• | 4.00 |
| 5299-1969 | Methods of sampling and | tests for d | ye interme | diates | ••• | ••• | 12.00 |
| 5438-1969 | Nitrobenzene-m-sulphonic | acid (sod | lium salt) | ••• | ••• | ••• | 3.50 |
| 6258-1971 | o-Nitroanisole | ••• | ••• | ••• | ••• | ••• | 2.00 |
| 6259-1971 | Anthraquinone, technical | | ••• | ••• | ••• | ••• | 2.50 |
| 6260-1971 | p-Nitroanisole | ••• | ••• | ••• | ••• | ••• | 2.00 |
| 6264-1971 | J-Acid | ••• | *** | * | ••• | ••• | 2.00 |
| 6265-1971 | Quinizarine, technical | ••• | ••• | ••• | ••• | ••• | 2.50 |
| 6266-1071 | 1.4 Diaminoanthmassinos | a tachnica | .1 | | | | 2.50 |

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| Brushware | fruits |
| Ceramicware, enamelware and labora- | Painters' materials (miscellaneous) |
| tory porcelain | |
| Chemical hazards and safety | Paper and its products |
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| Chemicals, organic (miscellaneous) | Perfumery materials, natural and |
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| Industrial gases | products |
| Inks and allied products | Thermal insulation materials |
| Laboratory glassware, thermometers | Thinners and so'vents |
| and related apparatus | Varnishes and lacquers |
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| dressings | Unclassified * * |
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AMENDMENT NO. 1 DECEMBER 2003 TO

IS 6260: 1971 SPECIFICATION FOR p-NITROANISOLE

(Page 3, clause 0.2, Structural Formula) — Insert the following below the structural formula:

'(CAS Registry No. 100-17-4)'

(Page 4, Table 1) — Substitute the following table for the existing:

Table 1 Requirements for p-Nitroanisole

| SI No. | Characteristic | Requirement | Methods of Test, Ref to | | |
|--------|--|-------------|-------------------------------|---------------------------------|--|
| | | | Clause of This Standard | Clause No of IS 5299 2001 | |
| (1) | (2) | (3) | (4) | (5) | |
| 1) | Crystallization point, ⁿ C | 51 2-52 0 | | 8 | |
| u) | Nitro compound content, percent by mass (expressed as C7H7NO ₃ , Mol Wt 153), Min | 98 5 | | 15 | |
| 111) | Matter insoluble in methanol, percent by mass | 0 2 | | 113 | |
| IV) | Presence of impurities, Max a) p-Nitrochlorobenzene (PCNB) | 02 | Annex A | 17 3 | |
| | b) o-Nitroanisole c) p-Nitrophenol | 0 2 0 2 | | | |

(Page 5, clause 4.3) — Insert the following Annex at the end:

'ANNEX A (Table 1)

THIN LAYER CHROMATOGRAPHIC ANALYSIS FOR DETERMINATION OF IMPURITIES

A-1 PROCEDURE

Impurities are determined by thin layer chromatography. Reference may be made to 'IS 5299: 2001 Methods of sampling and tests for dye intermediates' for details of TLC test method to be followed. However necessary details of test conditions are given here for guidance only.

Amend No. 1 to IS 6260: 1971

| 1 | Product name | p-Nitroanisole | | |
|--|---|---|--|--|
| 2 | Sample solution (on 100 % basis) | 2% Solution acetone | | |
| 3 | Application/sample volume for spotting | 5 μl (for sample) 2 μl and 4 μl (for impurities) | | |
| 4 | Standard | Reference standard | | |
| 5 | Test substanace for impuirties | 1) PCNB 2) o-nitroanisole 3) o-Nitro phenol (0 05 % solution in acetone) | | |
| 6 | Plate type | Silica gel G | | |
| 7 | Eluent | P E (60-80 C) Ether 70 30 | | |
| 8 | Elution time | (Ammonia atrh -saturated-twice run, 1h) | | |
| 9 | Temperature | 25 ± 5°C | | |
| 10 Detection spray | | (*)SnCl ₂ solution + PDAB solution | | |
| 11 | Evaluation | Semi quantitative | | |
| 12 | Approx Rf value — Main band —Impurities | p-Nitro anisole Rf 0 65 P- Nitrochlorobenzene (PCNB) o-Nitro anisole Rf 0 55 p- Nitrophenol Rf 0 15 | | |
| (*)SnCl2 solution 10 % solution in (1 1)water + 5N | | • | | |

(PCD 11)